

PTO 09-0929

CC=JP
DATE=20020726
KIND=KOKAI
PN=02208125

Co-Cr-Pt BASED TARGET MATERIAL AND MAGNETIC RECORDING MEDIUM
[Co-Cr-Pt KEI TAAGETTO ZAI OYABI JIKI KIROKU BAITAI]

TOMONORI UENO et al.

UNITED STATES PATENT AND TRADEMARK OFFICE
WASHINGTON, D.C. NOVEMBER 2008
TRANSLATED BY: SCHREIBER TRANSLATION, INC.

PUBLICATION COUNTRY (10) : JP

DOCUMENT NUMBER (11) : 02208125

DOCUMENT KIND (12) : KOKAI

PUBLICATION DATE (43) : 20020728

APPLICATION NUMBER (21) : 2001-000753

APPLICATION DATE (22) : 20010501

INTERNATIONAL CLASSIFICATION (51) : G 11 B 5/64
C 23 C 14/34
H 01 F 10/16
C 22 C 19/07

PRIORITY COUNTRY (33) :

PRIORITY NUMBER (31) :

PRIORITY DATE (32) :

INVENTOR(S) (72) : Tomonori Ueno
Hideo Murata
Shigeru Taniguchi

APPLICANT(S) (71) : Hitachi Metals Ltd.

DESIGNATED CONTRACTING STATES (81) :

TITLE (54) : Co-Cr-Pt Based Target
Material and Magnetic
Recording Medium

FOREIGN TITLE [54A] : Co-Cr-Pt Kei Taagetto Zai
oyobi Jiki Kiroku Baitai

[Claims]

[Claim 1]

A Co-Cr-Pt system target material, wherein the arithmetic average roughness Ra in an erosion part of a sputtering surface of a target material is less than 1.50 μm .

[Claim 2]

The Co-Cr-Pt system target material in Claim 1, which contains at least one or two element types chosen from B, Ti, Zr, Hf, V, Nb, Ta, Mo, W, Mn, Re, Ru, Os, Rh, Ir, Ni, Pd, Cu, Ag, Au, and C.

[Claim 3]

The Co-Cr-Pt system target material in Claim 1 or 2, which comprises $5 \leq \text{Cr} \leq 30$ at%, and $5 \leq \text{Pt} \leq 30$ at%.

[Claim 4]

The Co-Cr-Pt system target material in any of Claims 1 to 3, which comprises $0 < B \leq 25$ at%.

[Claim 5]

The Co-Cr-Pt system target material in any of Claims 1 to 4, which comprises
 $0 < (\text{Ti} + \text{Zr} + \text{Hf} + \text{V} + \text{Nb} + \text{Ta} + \text{Mo} + \text{W} + \text{Mn} + \text{Re} + \text{Ru} + \text{Os} + \text{Rh} + \text{Ir} + \text{nickel} + \text{Pd} + \text{Cu} + \text{Ag} + \text{Au} + \text{C})$
 ≤ 40 at%.

[Claim 6]

The Co-Cr-Pt system target material in any of Claims 1 to 5, wherein the average grain diameter of a matrix is 40 μm or less.

[Claim 7]

The Co-Cr-Pt system target material in any of Claims 1 to 6, wherein the purity is 3N or more.

[Claim 8]

The Co-Cr-Pt system target material in any of Claims 1 to 7, wherein the Fe content is 50 ppm or less.

[Claim 9]

The Co-Cr-Pt system target material in any of Claims 1 to 8, wherein the oxygen content is 20 ppm or less.

[Claim 10]

A magnetic recording medium, wherein at least one or more layers of Co-Cr-Pt system thin membrane which uses the Co-Cr-Pt system target material in any of Claims 1 to 9 is formed on a nonmagnetic substrate.

[Detailed Description of the Invention]

[0001]

[Technical Field]

This invention relates to the Co-Cr-Pt system target material which is used to form a magnetic membrane of a magnetic recording medium used for magnetic disc devices and the magnetic recording medium produced using said target material.

[0002]

[Related Art]

Conventionally, the Co system magnetic membrane has been developed to enable high-density magnetic recording, and Cr and Pt have been added to the Co system magnetic membrane. Furthermore, recently it is reported in J. Appl. Phys. 84, 6202 (1998)., etc. that the magnetic property is significantly improved by adding B to the Co-Cr-Pt system magnetic membrane.

/2

[0003]

As a technique for producing this Co-Cr-Pt system magnetic membrane, a sputtering technique, etc. can be used as described in the above literature, etc. In the sputtering technique, the target material is required as the supply source for the membrane composition. The target material used to form the above Co-Cr-Pt system magnetic membrane is an ingot which is normally dissolved, cast, and hot-rolled before machining and the surface finish thereof is performed by cutting such as lathing, etc.

[0004]

[Problems to Be Solved]

Conventionally, the surface finish of the Co-Cr-Pt system target material has been performed by cutting. Since expensive Pt is added, it is easier to collect it as ground powder from the viewpoint of recycling. Also, through the surface finish by cutting, abrasive grains sink into the target material surface,

which was considered to deteriorate the membrane property. As a result, the arithmetic average roughness Ra of the Co-Cr-Pt system target material produced by the conventional cutting was 1.50 μm or more. However, a problem occurred with a magnetic membrane which was formed using the target material produced by such a technique, i.e., dispersion of the magnetic property including the coercitivity and squareness ratio of the magnetic membrane produced. The purpose of this invention is to provide the Co system target material which has less magnetic property dispersion described above and has excellent membrane property.

[0005]

[Means for Solving the Problem]

After the inventors investigated the effect of the arithmetic average roughness Ra in the erosion part of the sputtering surface of the Co-Cr-Pt system target material on the magnetic property, they discovered that the arithmetic average roughness Ra in the erosion part of the sputtering surface of the Co-Cr-Pt system target material significantly affects the membrane composition, particularly the amount of Pt in the membrane. It became clear that the membrane composition, particularly the amount of Pt in the membrane as well as the dispersion thereof have a significant effect on the magnetic property of the magnetic recording medium including the coercitivity and squareness ratio of the magnetic membrane.

[0006]

With an alloy target material which consists of combination of an element with a large specific gravity difference such as the Co-Cr-Pt system target material, directivity may arise on the sputtered particles at the time of sputtering particularly on an ejection angle of an element with a large specific gravity, which makes the dispersion of the membrane composition larger, thereby varying the magnetic property such as the coercitivit or squareness ratio of the magnetic membrane of the magnetic recording medium. However, it was not thought to significantly

/3

affect the surface roughness.

[0007]

The surface roughness in the erosion part of the sputtering surface of the target material is not related to the essential problem of the ejection angle of the sputtered particles described above. However, as the surface roughness becomes coarser, the inclination angle of the target surface becomes larger, with the surface area changing, and additionally the incidence angle of an acceleration ion such as an Ar ion to the target surface, etc. changes as well, thereby making the directivity of the ejection angle of the sputtering grains more notable.

[0008]

The inventors studied the relation between the sputtering membrane property of the Co-Cr-Pt system target material and the surface roughness in the erosion part of the sputtering surface, and discovered that the dispersion in the magnetic property could be reduced through managing the arithmetic average roughness Ra which showed the surface roughness, thereby reaching this invention.

[0009]

In other words, this invention is the Co-Cr-Pt system target material, wherein the arithmetic average roughness Ra in the erosion part of the sputtering surface of the target material is less than 1.50 μm .

[0010]

The composition of the Co system target material of this invention preferably comprises $5 \leq \text{Cr} \leq 30$ at% and $5 \leq \text{Pt} \leq 30$ at% with the rest essentially comprising Co, and furthermore it may contain $0 < \text{B} \leq 25$ at%. It may contain $0 < (\text{Ti} + \text{Zr} + \text{Hf} + \text{V} + \text{Nb} + \text{Ta} + \text{Mo} + \text{W} + \text{Mn} + \text{Re} + \text{Ru} + \text{Os} + \text{Rh} + \text{Ir} + \text{nickel} + \text{Pd} + \text{Cu} + \text{Ag} + \text{Au} + \text{C}) \leq 40$ at% of one or two types or more of elements chosen from Ti, Zr, Hf, V, Nb, Ta, Mo, W, Mn, Re, Ru, Os, Rh, Ir, Ni, Pd, Cu, Ag, and C.

[0011]

Furthermore, as for the Co-Cr-Pt system target material of this invention, it is preferable that the average grain diameter

of the matrix is 40 μm or less, the purity is 3N or more, the Fe content is 50 ppm or less, and the oxygen content is 20 ppm or less. It is possible to provide a stable production of the magnetic recording medium through forming the Co-Cr-Pt system magnetic membrane by using the Co-Cr-Pt system target material of this invention.

[0012]

[Embodiments]

The greatest characteristic of this invention is that the arithmetic average roughness Ra in the erosion part of the sputtering surface of the target material Co-Cr-Pt system target material is less than 1.50 μm .

[0013]

The Co-Cr-Pt system target material of this invention allows a steady production of the magnetic recording medium by making the arithmetic average roughness Ra in the erosion part of the sputtering surface of the target material 1.50 μm or less, thereby keeping the Pt amount in the membrane equivalent to the target composition or more, while reducing the composition dispersion as well as controlling the magnetic property dispersion including the coercitivitiy and squareness ratio of the magnetic membrane for the magnetic recording medium formed. The arithmetic average roughness Ra is preferably less than 1.00 μm , and more preferably less than 0.50 μm . However, when the

surface roughness in the erosion part circumference, etc. in the sputtering surface is made coarser intentionally so that the scaffolding does not peel off, the surface roughness in the roughened region is not included in the specification of the arithmetic average roughness Ra of this invention.

[0014]

As a specific technique for making the arithmetic average roughness Ra of this invention less than $1.50 \mu\text{m}$, the technique for performing dry etching on the surface of the target material which underwent cutting process to smooth and refine/miniatrize the arithmetic average roughness Ra, for example, is effective. However, the surface finishing technique in this invention is not limited to the technique described above. Although the productivity declines, it can be achieved by lowering the feeding speed of a cutting knife, i.e., byte feeding speed at the time of the cutting process, or selecting the type of the cutting knife. Furthermore, a technique for eliminating ground grains entered into the target surface from the post-finish dry etching can be employed.

[0015]

The preferable composition range of the Co-Cr-Pt system target material of this invention is $5 \leq \text{Cr} \leq 30 \text{ at\%}$ and $5 \leq \text{Pt} \leq 30 \text{ at\%}$ with the remainder part essentially comprising Co. Cr has an

effect to segregate to the grain boundary in the membrane, thereby making the grain boundary nonmagnetic to divide the ferromagnetic Co grain magnetically. Since an addition of less than 5 at% does not make adequate magnetic division, while an addition exceeding 30 at% lowers the magnetization excessively, $5 \leq \text{Cr} \leq 30$ at% is preferable.

[0016]

Pt improves the magnetic anisotropy through solid solution into Co and effectively raises the coercitivity of the membrane. In order to increase the coercitivity, the addition of 5 at% or more has a significant effect, while the addition exceeding 30 at% significantly lowers the magnetic property such as magnetic anisotropy, etc. of Co's intrinsic property. Therefore, $5 \leq \text{Pt} \leq 30$ at% is preferable.

[0017]

It is effective to add B as the additive element to improve the magnetic anisotropy. B has an effect to segregate to the grain boundary in the membrane, thereby segregating the Pt element into the grain, and furthermore has an effect to segregate the nonmagnetic elements such as Cr, etc. to the grain boundary as well. However, since B is the element which promotes amorphousness wherein the addition of 25 at% destroys the

crystallinity of the membrane, thereby deteriorating the magnetic property, $0 < B \leq 25$ at% is preferable.

[0018]

Ti, Zr, Hf, V, Nb, Ta, Mo, W, Mn, Re, Ru, Os, Rh, Ir, Ni, Pd, Cu, Ag, Au, and C can be added as an additive element to improve the magnetic property. These elements are effective by adding a little amount, however, since the addition of a total amount exceeding 40 at% significantly destroys the magnetic property and crystallinity of the membrane, $0 < (Ti + Zr + Hf + V + Nb + Ta + Mo + W + Mn + Re + Ru + Os + Rh + Ir + Ni + Pd + Cu + Ag + Au + C) \leq 40$ at% is preferable.

[0019]

Furthermore, in the Co-Cr-Pt system target material of this invention, it is possible to further reduce the magnetic property dispersion such as the magnetic property of the magnetic membrane of the Co-Cr-Pt system sputtering membrane including the coercitivity and squareness ratio by making the average grain diameter of the matrix being 40 μm or less. Furthermore, 20 μm or less is preferable. With regard to the technique for making the average grain diameter of the matrix being 40 μm or less, it is effective to perform hot-plastic forming such as hot-rolling or hot forging to recrystallize the matrix, for example.

[0020]

It is possible to produce the target material with little dispersion in the average grain diameter of the matrix by controlling the hot plastic processing conditions such as the processing rate at the time of the hot plastic processing, for example. Specifically, since the dispersion becomes large due to the anisotropy at the time of the hot plastic processing if the processing rate is too high, while no recrystallization occurs if it is too low, the hot plastic processing is not effective.

When performing hot rolling as the hot plastic processing, cross rolling, for example is preferable since the structural anisotropy seldom appears by rolling. It is also possible to heat-treat before and after the hot plastic processing to control the structure.

[0021]

As for the Co-Cr-Pt system target material of this invention, the purity of 3N or more is preferable. Since particularly Fe and oxygen tend to blend into the target material, thereby easily degrading the membrane property, it is preferable to avoid contamination as much as possible. The membrane property decreases as the purity falls. Since when the impurity amount falls to 3N or less, the membrane property rapidly deteriorates, it is preferable to keep the purity 3N or

more, preferably 4N or more, and furthermore preferably 5N or more. Herein, the purity refers to a rate obtained by subtracting the total amount of elements excluding the main components from 100. 3N means 99.9%, 4N means 99.99%, and 5N means 99.999%.

[0022]

Since the membrane property deteriorates when Fe exceeds 50 ppm, it is set to 50 ppm or less, but it is preferable to set to 30 ppm or less, or still more preferably it is set to 10 ppm. Since the membrane property deteriorates when oxygen exceeds 20 ppm, it is set to 20 ppm or less, but it is preferable to set to 10 ppm or less. Herein ppm refers to mass ppm.

[0023]

[Embodiments of the Invention]

[Embodiment 1]

A target material of ϕ 180 mm \times 5 mm^t having the composition of Co-20Cr-10Pt-5B (at%) was produced as shown in Table 1. Vacuum melting and post-cast rolling were performed with Samples 1 - 10 to produce the target material. In Samples 1 - 5 a Co material having less Fe amount was used which underwent melting and casting, wherein the rolling rate at the time of the rolling was 50 %. In Samples 6 - 10 a Co material having more Fe amount was used which underwent melting and casting, wherein the

rolling rate at the time of the rolling was 25 %. In Samples 11 - 15, the target material was produced by powder sintering.

Furthermore, the surface roughness was changed by controlling the feeding rate of the byte of cutting process with the lathe at the time of the surface-finish processing.

[0024]

Table 1 shows the arithmetic average roughness Ra in the erosion part of the sputtering surface of the target material measured based on JIS-B0601, the matrix average grain diameter obtained by a cutting method on the microstructure from the sputtering surface with an optical microscope, Fe, and oxygen. The analysis result by GD-MS of Sample 1 is shown in Table 2 as an example. Furthermore, the surface roughness curve of Samples 2 and 3 of this invention and Sample 5 of the comparison example are shown in Figures 1, 2, and 3.

[0025]

By using the Al substrate which is NiP plated, a Cr base membrane was formed on the substrate under the condition of the substrate temperature 150°C, Ar pressure 0.66Pa, and the DC power 500W; a magnetic membrane was formed with the Co-20Cr-10Pt-5B (at%) target material produced under the various conditions shown in Table 1. In order to investigate the property dispersion of the magnetic membrane, the membrane formation substrate was produced with 1 hour interval from 1 hour to 4 hours of the

total membrane formation time. Table 3 shows the coercitivity H_c measured with a VSM (vibrating sample magnetometer).

[0026]

Table 3 shows that reducing the arithmetic average roughness R_a in the erosion part of the sputtering surface of the Co-Cr-Pt system target material, specifically, making it less than 1.50 μm as specified by this invention, the membrane property at the time of the sputtering membrane formation is improved and stabilized.

[0027]

[Table 1]

Sample	R_a (μm)	Average grain diameter (μm)	Fe (ppm)	Oxygen (ppm)	Remarks
1	0.27	22	42	8	This invention
2	0.48	22	42	8	This invention
3	0.68	22	42	8	This invention
4	1.03	22	42	8	This invention
5	1.52	22	42	8	Comparison example
6	0.25	51	76	10	This invention
7	0.42	51	76	10	This invention
8	0.71	51	76	10	This invention
9	1.10	51	76	10	This invention
10	1.55	51	76	10	Comparison example
11	0.29	5	45	64	This

					invention
12	0.38	5	45	64	This invention
13	0.65	5	45	64	This invention
14	1.06	5	45	64	This invention
15	1.53	5	45	64	Comparison example

[0028]

[Table 2]

/5

Element	Analysis value	Element	Analysis value	Element	Analysis value
L1	<928 ppt	As	<2.6 ppb	Nd	<4.4 ppb
Be	<764 ppt	Se	<22 ppb	Sm	<2.7 ppb
C	10.8 ppm	Br	<1.4 ppb	Eu	<969 ppt
N	1.96 ppb	Rb	<711 ppb	Gd	<2.4 ppb
O	<6.8 ppb	Sr	1.59 ppm	Tb	<360 ppb
Na	<1.0 ppb	Y	120 ppb	Dy	<2.0 ppb
Mg	<1.0 ppb	Zr	<847 ppb	Ho	<408 ppt
Al	23.4 ppm	Nb	52.6 ppb	Er	<1.6 ppb
S1	95.4 ppm	M0	<5.9 ppb	Tm	<4436 ppt
P	2.59 ppm	Ru	1.73 ppm	Yb	<3.4 ppb
S	9.28 ppm	Rh	798 ppb	Lu	<428 ppb
Cl	996 ppb	Rd	900 ppb	Hf	<1.2 ppb
K	38.2 ppb	Ag	<4.5 ppb	Ta	4.99 ppm
Ca	104 ppb	Cd	22.9 ppb	W	698 ppb
Sc	16.8 ppb	In	468 ppb	Re	31.9 ppb
Ti	8.69 ppm	Sn	12.4 ppm	Os	<3.1 ppb
V	163 ppb	Sb	<4.7 ppb	Ir	3.16 ppm
Mn	2.52 ppm	Te	<24 ppb	Au	243 ppb
Fe	45 ppm	I	<513 ppt	Hg	5.56 ppm
Ni	122 ppm	Cs	<513 ppt	Tl	46.9 ppm
Cu	10.1 ppm	Ba	<13 ppb	Pb	,5.4 ppb
Zn	1.33 ppm	La	<574 ppt	Bi	24.2 ppm
Ga	46.6 ppb	Ce	<431 ppt	Th	<43 ppb
Ge	<37 ppb	Pr	<413 ppt	U	<115 ppb

[0029]

[Table 3]

/ 6

Sample	Coercitivity (Oe) [(kA/m)]				Remarks
	1 hour	2 hours	3 hours	4 hours	
1	3163 [252]	3161 [252]	3167 [252]	3169 [252]	This invention
2	3155 [251]	3158 [251]	3150 [251]	3164 [252]	This invention
3	3142 [250]	3140 [250]	3151 [251]	3147 [251]	This invention
4	3136 [250]	3130 [250]	3145 [250]	3140 [250]	This invention
5	3066 [244]	3091 [246]	3077 [245]	3107 [247]	Comparison sample
6	3144 [250]	3150 [251]	3150 [251]	3158 [251]	This invention
7	3141 [250]	3142 [250]	3147 [251]	3150 [251]	This invention
8	3123 [249]	3127 [249]	3132 [249]	3138 [250]	This invention
9	3115 [248]	3121 [248]	3127 [249]	3132 [249]	This invention
10	3091 [246]	3061 [244]	3077 [245]	3097 [247]	Comparison sample
11	3150 [251]	3145 [250]	3151 [251]	3153 [251]	This invention
12	3151 [251]	3142 [250]	3152 [251]	3145 [250]	This invention
13	3120 [248]	3125 [249]	3131 [249]	3128 [249]	This invention
14	3111 [248]	3116 [248]	3133 [249]	3131 [249]	This invention
15	3074 [245]	3090 [246]	3081 [245]	3037 [240]	Comparison sample

[0030]

[Embodiments of the Invention]

[Embodiment 2]

The target of $\phi 180\text{mm} \times 5\text{mm}$ having the constitution shown in Table 4 was produced. The target was produced by vacuum melting and casting, and thereafter hot rolling with the rolling rate of 50%. Furthermore, the surface roughness was changed by controlling the feeding rate of the byte of the cutting process with the lathe at the time of the surface-finish processing.

[0031]

Table 5 shows the arithmetic average roughness R_a in the erosion part of the sputtering surface of the target material measured based on JIS-B0601, the matrix average grain diameter obtained by a cutting method on the microstructure from the sputtering surface with an optical microscope, Fe, and oxygen.

[0032]

By using the Al substrate which is NiP plated, a Cr base membrane was formed on the substrate under the condition of the substrate temperature 150°C, Ar pressure 0.66Pa, and the DC power 500W; a magnetic membrane was formed with the Co-Cr-Pt target material shown in Table 4. In order to investigate the property dispersion of the magnetic membrane, the membrane formation substrate was produced with 1 hour interval from 1 hour to 4 hours of the total membrane formation time. Table 6 shows the coercitivitiy H_c measured with a VSM (vibrating sample magnetometer).

[0033]

Table 6 shows that reducing the arithmetic average roughness Ra in the erosion part of the sputtering surface of the Co-Cr-Pt system target material, specifically, making it less than 1.50 μm as specified by this invention, the membrane property at the time of the sputtering membrane formation is improved and stabilized.

[0034]

[Table 4]

/7

Sample	Composition (%)
16	Co-20Cr-10Pt
17	Co-20Cr-10Pt
18	Co-20Cr-10Pt-2Ta
19	Co-20Cr-10Pt-2Ta
20	Co-20Cr-10Pt-2Ta-5B
21	Co-20Cr-10Pt-2Ta-5B
22	Co-20Cr-10Pt-5Ni-5B
23	Co-20Cr-10Pt-5Ni-5B
24	Co-20Cr-10Pt-2Cu-5B
25	Co-20Cr-10Pt-2Cu-5B

[0035]

**[Table 5]

Sample	Ra (μm)	Average grain diameter (μm)	Fe (ppm)	Oxygen (ppm)	Remarks
16	0.65	32	38	17	This invention
17	1.51	32	38	17	Comparison example
18	0.67	26	37	15	This invention
19	1.51	26	37	15	Comparison example

20	0.65	14	42	11	This invention
21	1.52	14	42	11	Comparison example
22	0.69	23	48	18	This invention
23	1.53	23	48	18	Comparison example
24	0.66	19	46	13	This invention
25	1.54	19	46	13	Comparison example

[0036]

**[Table 6]

Sample	Coercitivity (Oe) [(kA/m)]				Remarks
	1 hour	2 hours	3 hours	4 hours	
16	2939 [234]	2943 [234]	2950 [235]	2948 [235]	This invention
17	2853 [227]	2874 [229]	2908 [231]	2833 [226]	Comparison sample
18	3056 [243]	3055 [243]	3063 [244]	3067 [244]	This invention
19	2972 [237]	2978 [237]	2943 [234]	2954 [235]	Comparison sample
20	3168 [252]	3165 [252]	3174 [253]	3178 [253]	This invention
21	3057 [243]	3060 [244]	3082 [245]	3043 [242]	Comparison sample
22	3143 [250]	3139 [250]	3145 [250]	3150 [251]	This invention
23	3085 [246]	3093 [246]	3073 [245]	3102 [247]	Comparison sample
24	3166 [252]	3173 [253]	3171 [252]	3172 [252]	This invention
25	3051 [243]	3053 [243]	3076 [245]	3033 [241]	Comparison sample

[0037]

[Effects of the Invention]

The Co-Cr-Pt system target material of this invention
enables to make the Co-Cr-Pt system magnetic membrane used in

/8

the magnetic recording media for the magnetic disk devices etc.
excellent in the magnetic property, thereby making it
indispensable to produce the magnetic recording media.

[Brief Explanation of the Figures]

[Figure 1]

A part of the surface roughness curve of the erosion part
in Sample 2 of this invention.

/14

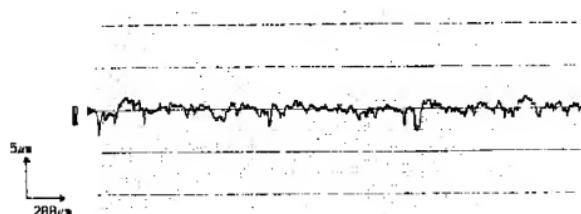
[Figure 2]

A part of the surface roughness curve of the erosion part
in Sample 3 of this invention.

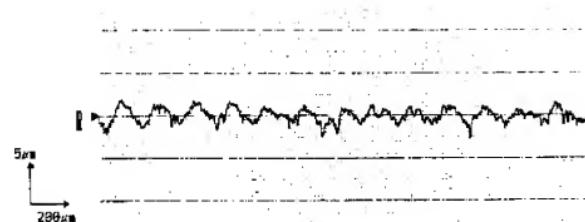
[Figure 3]

A part of the surface roughness curve of the erosion part
in Sample 5 of the comparison example.

[Figure 1]



[Figure 2]



[Figure 3]

